



IN THE U.S. PATENT AND TRADEMARK OFFICE

Applicant: Yoshinori Harada et al

Art Unit: 1755

Appl. No.: 10/718,575

Primary Examiner:

Elizabeth A. Borden

Filed: November 24, 2003

For: Highly Durable Silica Glass, Process and Apparatus for Producing Same, and Highly durable Glass Member and Apparatus Provided with Same

VERIFICATION OF TRANSLATION

I, Yukio Uchida, hereby declare the following:

I am knowledgeable in Japanese and English. I have reviewed the following two Japanese documents,

Document 1: Hiroyuki Matsunami "Semiconductor Technology", first edition, 6<sup>th</sup> print, published April 15, 1986 by K.K. Shoko-do, Japan

Document 2: Katsufusa Shono "100 Collections of Semiconductor Technology on Ultra-large Scale Integrated Circuit [II]", first edition, 3<sup>rd</sup> print, published July 10, 1983 by K.K. OHM, Japan

and believe the attached documents to be accurate partial translations thereof.

All statements made herein of my own knowledge are true and all statement made on information and belief are believed to be true. Further, these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: August 1, 2005

  
Yukio Uchida

Partial Translation of Hiroyuki Matsunami "Semiconductor Technology", first edition, 6<sup>th</sup> print, published April 15, 1986 by K.K. Shoko-do, Japan [Document 1]

[Page 109, lines 1-14 under Table 5.7.]

## 5.2 Purification of Semiconductor Material

Semiconductor is highly sensible for material, and its characteristics greatly vary depending upon the presence of a minor amount of impurities, if any. Therefore, to obtain a semiconductor having a controlled specific conductive type, for example, p-type or n-type semiconductor, it is necessary first to prepare pure material for semiconductor by thorough purification, and thereafter, incorporate a require amount of impurity. Elemental semiconductor materials such as silicon semiconductor and germanium semiconductor can be purified to an extent such that the impurity content is not larger about  $10^{-11}$  and  $10^{-10}$ , respectively. A purification method includes a chemical method and a physical method. A chemical method gives a purity of about 99.8% at most. A semiconductor material having an ultra-high purity is prepared by a physical method.

### 5.2.1 Chemical Purification

#### (1) Silicon (Si)

Metal-grade silicon (purity: 98-99%) prepared by reducing a  $\text{SiO}_2$  based silica stone is used as a raw material. Silicon (Si) is extracted as a halide or a hydride, and then chemically purified by distillation or other means. The main point of chemical purification lies in the chemical removal of boron (B) which is difficult to remove by a physical method.

Partial Translation of Katsufusa Shono "100 Collections of Semiconductor Technology on Ultra-large Scale Integrated Circuit [II]", first edition, 3<sup>rd</sup> print, published July 10, 1983 by K.K. OHM, Japan [Document 2]

[Page 16, left column, lines 1-12]

### 13 Contamination by CVD Apparatus

Unexpected troubles often arise in the process due to the fact that a CVD (chemical vapor deposition) apparatus used is contaminated with impurity elements of group III such as boron or impurity elements of group V such as phosphorus or arsenic. These troubles are very difficult to find, and therefore, maintenance of the apparatus must be carefully carried out so as to avoid such troubles.

Experimentally most offensive contamination is caused by boron. In the case when  $H_2$ - or  $N_2$ -based 5%  $B_2H_6$  causes deposition of BSG (boro-silicate glass) or polycrystalline silicon, 5%  $B_2H_6$  is often used as a doping gas. 5%  $B_2H_6$  is used as a gas of low concentration diluted with a large amount of carrier gas such as  $N_2$  or  $H_2$ . After completion of experiments,  $B_2H_6$  is thoroughly flushed and disposed. But, residual  $B_2H_6$  in a pipe often causes contamination. It is free from contamination and safe to use  $B_2H_6$  as an admixture gas having a concentration of 1% rather than 5%.

[Page 16, left column, lines 25-32]

To check whether a pipe is contaminated with boron or not, a silicon wafer is placed on a heating base plate where  $Si_3N_4$  having a thickness of about 400 Å is deposited thereon to prepare a test specimen. The test specimen is heat-treated at a temperature of 1,200°C in a nitrogen gas for 1 hour in a diffusion furnace. The formation of a diffusion layer is checked depending upon the particular p-type or n-type semiconductor. In the case when a diffusion layer is not formed on any of p-type and n-type

silicon wafers. The magnitude of contamination with boron assigned is regarded as not larger than  $10^{15} \text{ cm}^{-3}$ . In the case when a diffusion layer is formed, the resistivity is measured by a 4 point probe method, and the diffusion depth  $x_j$  is measured from spherical drill and stain. From the measurement values, concentration ( $C_s$ ) of impurities on the surface of diffusion layer, diffusion coefficient ( $D$ ) and the total amount ( $Q$ ) of impurities are determined.

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## 5.2 半導体材料の精製

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表 5.7 代表的な有機半導体とその性質

| 有機化合物                  | 導電率 <sup>1</sup><br>$\sigma$ ( $\text{Sm}^{-1}$ ) | 活性化エネルギー<br>$E$ (eV) | 光学的<br>禁帯幅<br>(eV) |
|------------------------|---|----------------------|--------------------|
| 芳香族多環系<br>ナフタレン        | $10^{-3}$   | 1.65                 | 3.8                |
| アントラセン                 | 8   | 0.83                 | 3.0                |
| ペトラセン                  | 10  | 0.85                 | 3.6                |
| フタロシアノン                | $5 \times 10^3$                                   | 0.9                  |                    |
| キレート化合物<br>銅フタロシアノン    | $10^4$  | 0.9                  |                    |
| ポリアセチレン                | $10^{-4} \sim 10^{-3}$                            | 0.8                  |                    |
| 鎖状高分子<br>ポリフェニルアセチレン   |   | 1.4~2.8              |                    |
| ポリアクリルニトリル             | $(10^{-2} \sim 10^{-7})$                          | 0.64                 |                    |
| 分子間化合物<br>テトラウアノキノジメタン | $(10^4)$  |                      |                    |

<sup>1</sup>  $\sigma = qn \exp(-E/kT)$  における  $\sigma_0$  ( ) 内は 20°C での値

## 5.2 半導体材料の精製

半導体は構造敏感であって、わずかの不純物でも存在すればその特性が著しく変わる。したがって、伝導形を制御してp形やn形を得る場合には、まず十分に精製して真性半導体にし、その上で必要量の不純物を添加する。SiやGeの元素半導体では、不純物密度がそれぞれ  $10^{-11}$ ,  $10^{-9}$  以下程度にまで精製することができる。精製には化学的方法と物理的方法がある。化学的方法では純度は99.8%程度しか上がらない。超高純度の材料は物理的精製法を用いて製作される。

## 5.2.1 化学的精製

## (1) シリコン (Si)

珪石 ( $\text{SiO}_2$  が主成分) を還元して得られる金属級 Si (純度 98~99%) を原料として用いる。Si はハロゲン化物、あるいは水素化物にして抽出、蒸留などにより化学的に精製する。物理的精製法で除去することが困難なボロン (B) を化学的に除去することに重点を置いている。高純度化されたこれらの Si 化合物を熱

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専門 半導体材料

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